REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY *(Leave blank)*

2. REPORT DATE
September 1, 1994

3. REPORT TYPE AND DATES COVERED
Final 15 Jun 91 - 14 Jun 94

4. TITLE AND SUBTITLE
SUPERPLASTIC CERAMICS

5. FUNDING NUMBERS
DAAL03-91-G-0172

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
U. S. Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709-2211

10. SPONSORING/MONITORING AGENCY REPORT NUMBER
ARO 28761.1-MS

11. SUPPLEMENTARY NOTES
The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

Superplasticity is a newly observed phenomenon in ceramics, with the first publication appearing in 1986. The current program has centered on an experimental study of superplasticty in polycrystalline iron carbide. This material has been made superplastic by utilization of two processing methods: powder processing and ingot-processing. In both cases the end microstructure is a continuous phase of ultra-fine grained iron carbide. The ultra-fine grained powder processed material was successfuly gas-pressure blow-formed into a spherical shaped object, and its deformation followed the predicted behavior of a high strain-rate sensitive material that is controlled by grain-boundary sliding. Thermo-mechanical processing routes were developed to refine the coarse as-cast ingot microstructure. Structural refinement is a result of creation of strain-free regions by carbon dissolution from high strain energy subgrain boundaries and slip bands. It is proposed that thermo-mechanical processing of ingot-cast eutectic-composition oxide ceramics to achieve a superplastic structure is feasible.

14. SUBJECT TERMS
Superplasticity, ceramics, iron carbide, fine-grained materials, atomized powders, ingot-processing, tensile elongations, fracture

15. NUMBER OF PAGES
9

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	UL

Final Report

SUPERPLASTIC CERAMICS

September 1, 1994

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A. STATEMENT OF THE PROBLEM STUDIED

The two principal objectives of this program are (1) to develop processing methods for achieving stable fine-grained carbide base materials in order to obtain superplastic behavior, and (2) to evaluate and understand the factors influencing the tensile ductility of fine-grained ceramics that exhibit high strain-rate sensitivity.

B. SUMMARY OF THE MOST IMPORTANT RESULTS

The study was centered on the superplastic behavior of ceramic-base materials. The experimental effort was primarily on developing novel procedures for attaining fine structures in iron carbide type materials, and on studying their mechanical behavior at elevated temperature. A major discovery is that ceramic materials are amenable to high tensile elongations, if the appropriate fine structure is developed , and if the

appropriate rate of straining and temperature is selected. In the following, a general description of superplasticity is given, including its potential importance in industrial applications. Three figures are included which summarize well the major findings and conclusions in the present research program. The specific publications that have resulted are summarized in Section C. The contributors to the program are listed in Section D.

Superplasticity and superplastic metals and ceramics

Superplastic polycrystalline materials are extraordinarily stretchable, with an enhanced ability to flow plastically, much like molasses, molten glass or other viscous fluids. Most conventional crystalline materials can be stretched only about 50 percent beyond their original length. Some superplastic crystalline materials can be stretched like taffy, 1,000 percent and more. The scientific basis for achieving superplasticity is the development of ultra-fine grains, typically less than 10 μm (.0l mm). An example of such a super-stretchable fine-grained material, developed at Stanford, is shown in the upper left inset of Figure 1.

Superplasticity allows solid materials to flow so easily into molds that they can be used to form extremely complex shapes, such as gears, in a single procedure. This eliminates the need for expensive welding and machining steps now needed to manufacture intricate shaped parts. Machining a part into its final shape means wasted time, material and money. Up to 50 percent of the original quantity of material winds up as shavings or other scrap. Superplastic materials thus offer the prospect of substantial economic savings for American industry. Because of this unique quality, interest in this field is growing, and this is well

documented by the increasing number of publications with the years. This is shown in Figure 1.

Of special significance in the graph (Figure 1) is the the new field of superplastic ceramics. The first publication on superplastic ceramics was in 1986, and in 1991, thirty publications appeared on this subject. At Stanford, our first publication on superplastic ceramics, under the sponsorship of the Army Research Office (ARO), was in 1989. Since then, sixteen papers have been written and submitted for publication based on the ARO sponsored program. Nine of these papers, representing the past three years of activity, are listed in Section C. Our emphasis at Stanford has been to develop unique processing procedures to achieve ultra-fine grained structures in ceramic iron carbide, and to evaluate their superplastic properties at elevated temperature. An example of a gas-pressure blow-formed component of fine-grained iron carbide is given in the top right inset of Figure 1. This demonstration attests to the high formability of fine-grained ceramics.

The fine-grained iron carbide material, described above, was prepared by the traditonal powder processing approaches used in preparing structural ceramics. At Stanford, we have recently initiated an alternative route to achieve fine structures involving thermal-mechanical working of an ingot material. A major advantage of the newly proposed route is that fine grains can be developed containing impurity-free grain boundaries. The ingot-processing route has not been used in traditional ceramic processing because it is believed that ceramics are brittle at all temperatures. We have shown that this is not true when two phase ceramic-base materials are used. Figure 2 illustrates the mechanical working steps developed to generate a superplastic microstructure in a

cast-ingot of the iron carbide material (80% iron carbide-20% iron). The five-step sequence of events shown involve the hot pressing, canning and rolling of the iron carbide material. It can be noted that the initial structure which consists of coarse proeutectic carbides (the first ones to form upon solidification) is refined after hot pressing (step 2) and further refined after extensive rolling (step 5). Structural refinement is a result of creation of strain-free regions by carbon dissolution from high strain energy subgrain boundaries and slip bands. The mechanically processed ingot material was shown to be superplastic (75% elongation). Higher elongations can be achieved by additional mechanical working which will further refine the microstructure.

These encouraging results suggest that oxide ceramics can be made superplastic by ingot processing when a eutectic-composition binary oxide system is selected. Figure 3 illustrates and explains the basis for the expected success in a eutectic composition ZrO_2-Al_2O_3 material. It is believed that the ingot-processing route will offer a potentially economical alternative to the powder-processing route because fewer processing steps are required.

C. LIST OF ALL PUBLICATIONS

1. W.J. Kim, J. Wolfenstine, O.A. Ruano, G. Frommeyer and O.D. Sherby, " Processing and Superplastic Properties of Fine-Grained Iron Carbide", Metall. Trans. 23A, 527-535, 1992.

2, Woo Jin Kim, "Superplastic Ceramics (with an emphasis on iron carbide materials)", Ph.D. Dissertation, Stanford University, Stanford, CA. 94305, June 1993.

3. Woo-Jin Kim, J. Wofenstine and Oleg D. Sherby, "Ingot Processing as an Alternative to Powder Processing for Achieving Superplasticity in Ceramics", Journal of the Ceramic Society of Japan, 102, 835-843, 1994.

4. J. Wittenauer, W.J. Kim and O.D. Sherby, "Superplastic Gas-pressure Deformation of Iron Carbide Sheet", accepted for publication, Materials Science and Engineering, 1994.

5. Woo-Jin Kim and Oleg D. Sherby, "Superplasticity in Ingot-Processed Iron Carbide", Proceedings of the International Conference on Superplasticity in Advanced Materials", (ICSAM-94), May 24-26, 1994, Moscow, Russia, published by Trans. Tech Publications Ltd. CH-4714, Aedermannsdorf, Switzerland.

6. Oleg D. Sherby, T.G. Nieh and J. Wadsworth, "Overview on Superplasticity Research on Small-Grained Materials", Proceedings of the International Conference on Superplasticity in Advanced Materials, (ICSAM-94), May 24-26, 1994, Moscow, Russia, published by Trans. Tech Publications Ltd, CH-4714, Aedermannsdorf, Switzerland.

7. Woo-Jin Kim and Oleg D. Sherby, "Tensile Ductility Behavior of Fine-grained Alumina at Elevated Temperature", submitted for publication, May 1994, J. Amer. Cer. Soc.

8. C.K. Teo, O.A. Ruano, J. Wadsworth and O.D. Sherby, "Superplastic Behavior of a Ceramic-based Kappa/Alpha Fe-10Al-1.9C Material", J. Mater. Science, 1994, in press.

9. Woo-Jin Kim and Oleg D. Sherby, "Tensile Ductility Behavior of Superplastic Ceramics and Ceramic Composites" (a review) , in Key Engineering Materials - Ceramic Matrix Composites, Eds. Erian Armanios, Yiu-Wing Mai and Fred H. Wohlbier, Trans. Tech Publications Ltd, CH-4714, Aedermannsdorf, Switzerland, June 1995.

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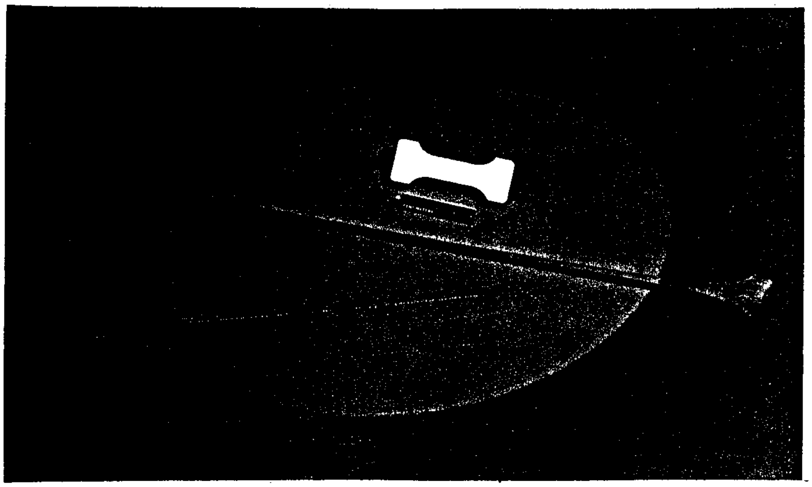
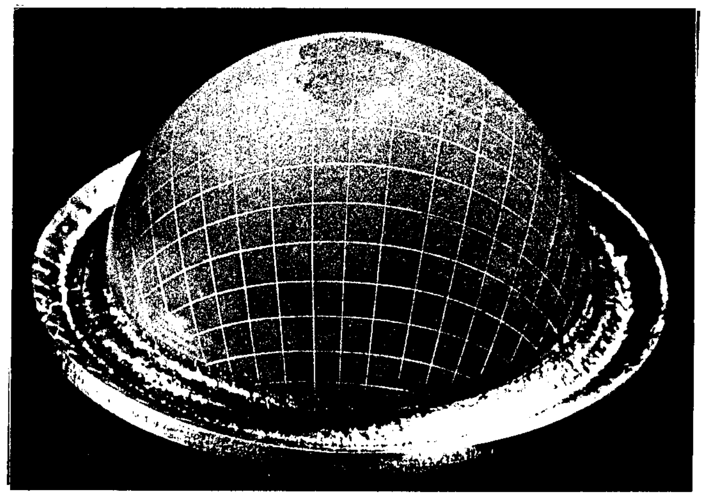
E. ACKNOWLEDGEMENTS

This program was sponsored and monitored by the Army Research Office under Contract DAAL-03-88-K-0056. The authors are grateful for the support, encouragement and technical suggestions from the contract monitors, Drs. Wilbur Simmons, Andrew Crowson and Edward Chen.

HIGH TENSILE ELONGATION ILLUSTRATING SUPERPLASTICITY IN A FINE-GRAINED MATERIAL.



GAS PRESSURE SUPERPLASTIC BLOW FORMING OF AN IRON CARBIDE CERAMIC MATERIAL



PUBLICATIONS AS A FUNCTION OF TIME ILLUSTRATING GROWTH OF THE NEW FIELD OF SUPERPLASTIC CERAMICS

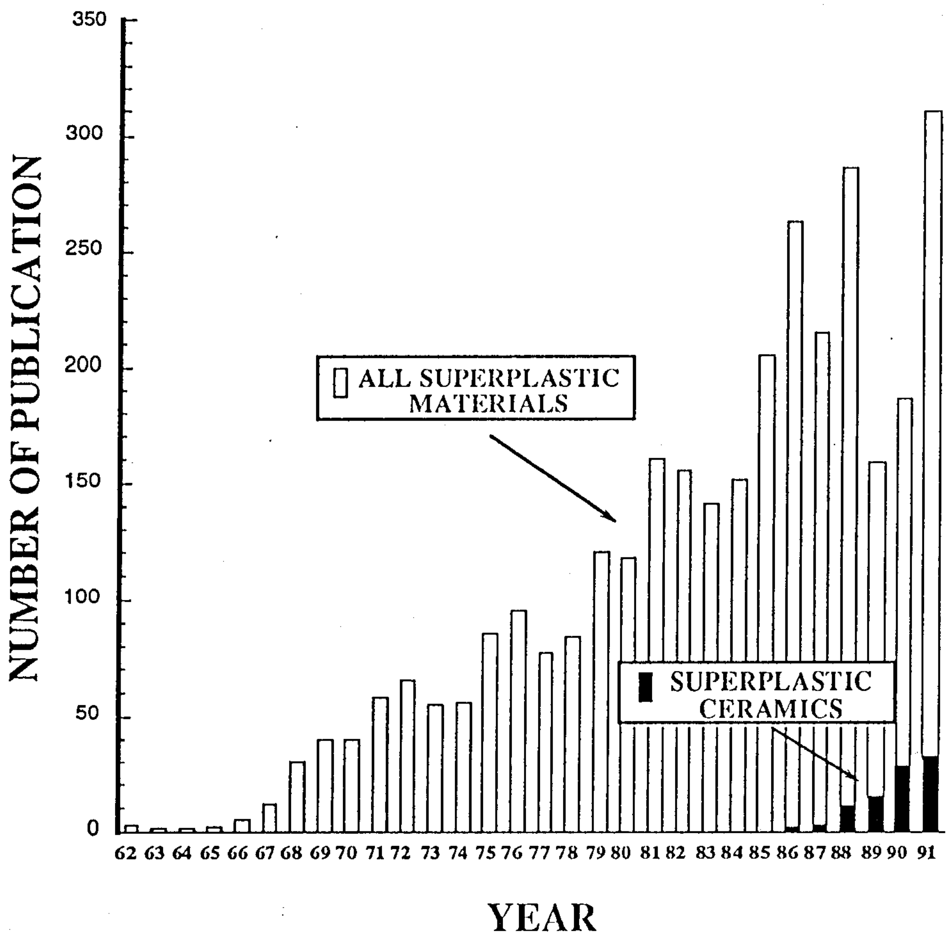


FIGURE 1.

ILLUSTRATION OF MECHANICAL WORKING STEPS TO GENERATE A SUPERPLASTIC MICROSTRUCTURE IN A CAST-INGOT OF IRON CARBIDE

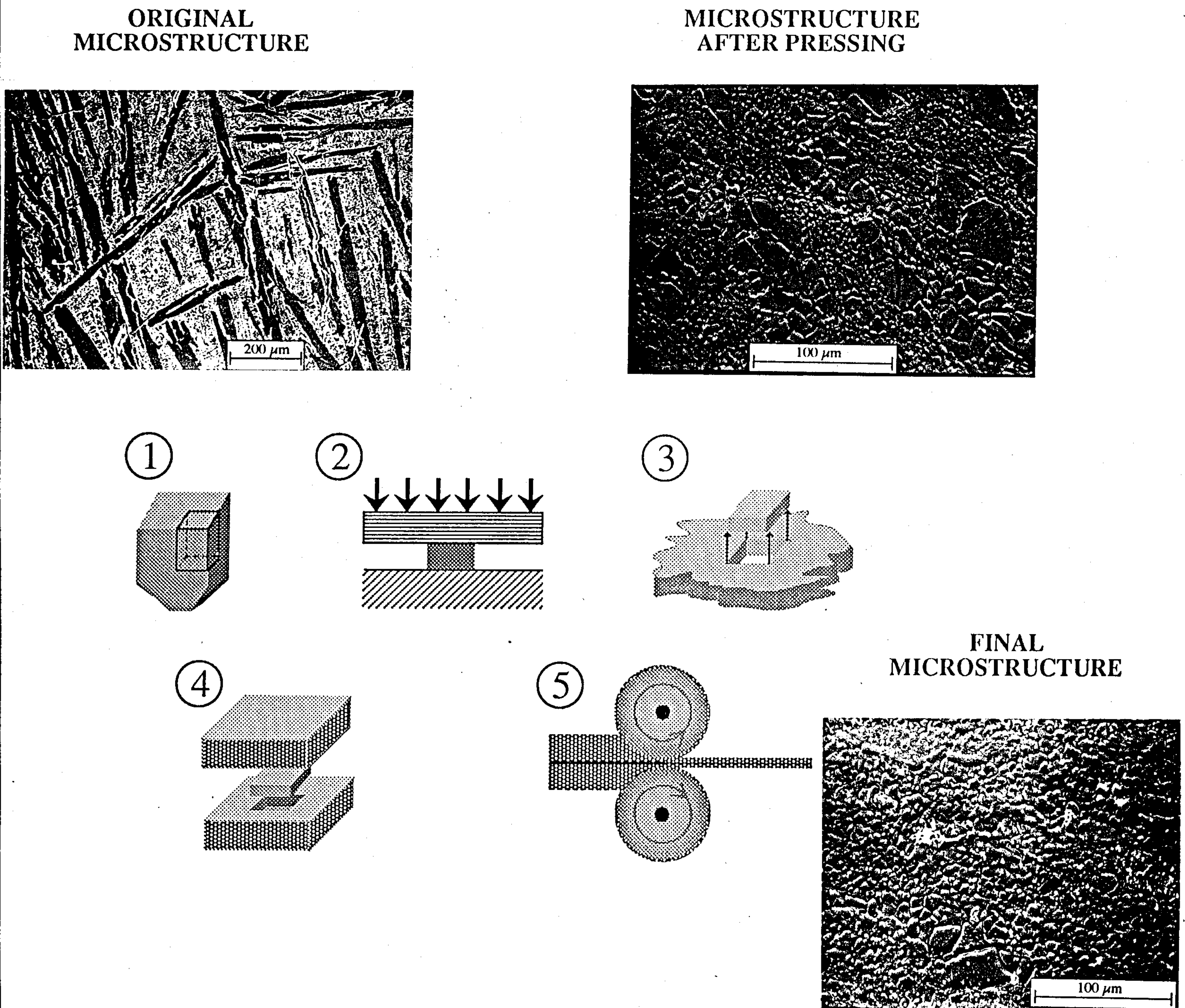
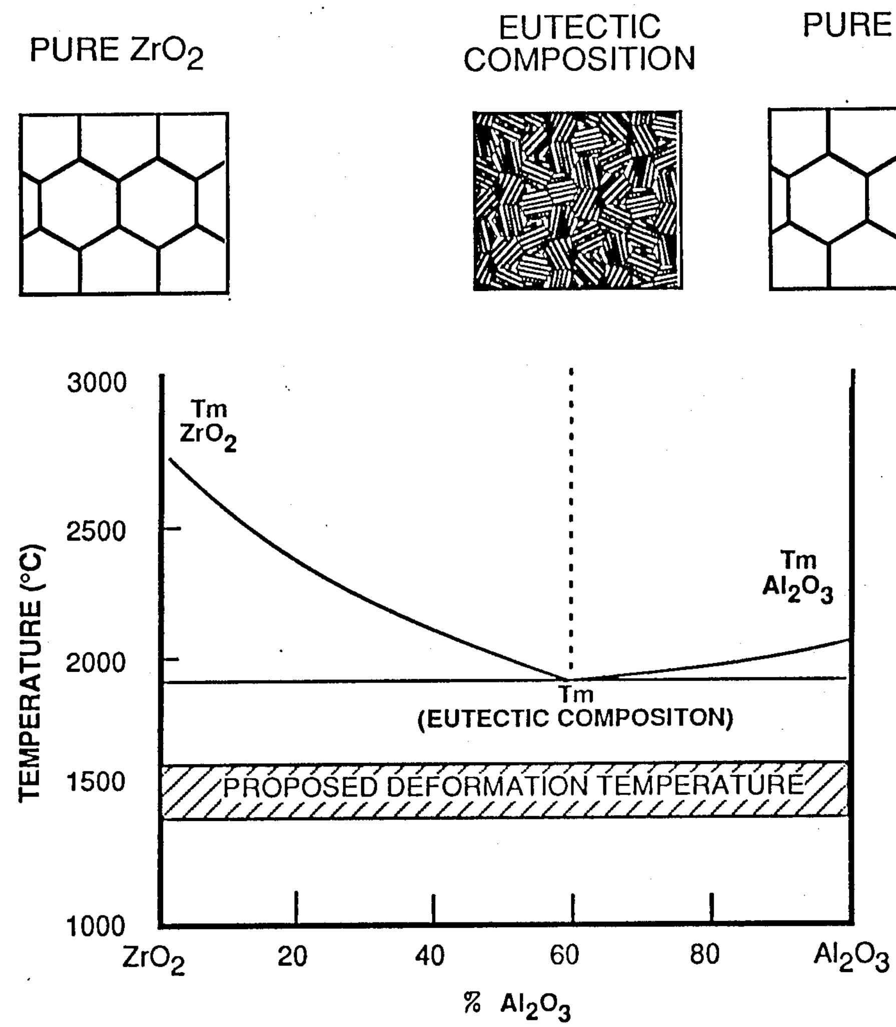


FIGURE 2.



PROPOSED: DEVELOPMENT OF SUPERPLASTIC CERAMICS BY INGOT-PROCESSING OF A EUTECTIC-COMPOSITION BINARY OXIDE SYSTEM.

SUCCESS EXPECTED BECAUSE:

1) INITIAL STRUCTURE CONSISTS OF FINE ALTERNATING PLATES OF EACH OXIDE PHASE.

2) LARGE NUMBER OF INTERPHASE BOUNDARIES REPRESENTS REGIONS OF HIGH DIFFUSION PATH SINCE MELTING POINT (Tm) IS LOW.

3) HOT MECHANICAL WORKING CAN BE READILY ACHIEVED BECAUSE RECOVERY PROCESSES ARE ENHANCED BY FINE INITIAL PLATE STRUCTURE AND BY PRESENCE OF MANY HIGH DIFFUSION PATHS.

4) INTERPHASE BOUNDARIES TYPICALLY HAVE LOW ENERGY BECAUSE THE MELTING TEMPERATURE IS LOW HENCE LIKELIHOOD OF CRACKING AT SUCH REGIONS IS MADE DIFFICULT (REQUIRES FORMATION OF TWO HIGH ENERGY SURFACES)

ADDITIONAL ADVANTAGE:

TEMPERATURE OF HOT WORKING IS RELATIVELY LOW COMPARED TO THE PURE OXIDE COMPONENTS, LEADING TO GREATER EASE OF PROCESSING.

FIGURE 3.